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PHASE CHANGES ON 4H AND 6H SIC AT HIGH TEMPERATURE OXIDATION

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ABSTRACT

PHASE CHANGES ON 4H AND 6H SIC AT HIGH TEMPERATURE OXIDATION. The oxidation on two silicon carbide contain 6H phase and contains 6H and 4H phases has been done. Silicon carbide is ceramic non-oxide with excellent properties that potentially used in industry. Silicon carbide is used in nuclear industry as structure material that developed as light water reactor (LWR) fuel cladding and as a coating layer in the high temperature gas-cooled reactor (HTGR) fuel. In this study silicon carbide oxidation simulation take place in case the accident in primary cooling pipe is ruptured. Sample silicon carbide made of powder that pressed into pellet with diameter 12.7 mm and thickness 1.0 mm, then oxidized at temperature 1000 ^{o}C, 1200 ^{o}C dan 1400 ^{o}C for 1 hour. The samples were weighted before and after oxidized. X-ray diffraction conducted to the samples using Panalytical Empyrean diffractometer with Cu as X-ray source. Diffraction pattern analysis has been done using General Structure Analysis System (GSAS) software. This software was resulting the lattice parameter changes and content of SiC phases. The result showed all of the oxidation samples undergoes weight gain. The 6S samples showed the highest weight change at oxidation temperature 1200 ^{o}C, for the 46S samples showed increasing tendency with the oxidation temperature. X-ray diffraction pattern analysis showed the 6S samples contain dominan phase 6H-SiC that matched to ICSD 98-001-5325 card. Diffraction pattern on 6S showed lattice parameter, composition and crystallite size changes. Lattice parameters changes had smaller tendency from the model and before oxidation. However, the lowest silicon carbide composition or the highest converted into other phases up to 66.85 %, occurred at oxidation temperature 1200 ^{o}C. The 46S samples contains two polytypes silicon carbide. The 6H-SiC phases matched by ICSD 98-016-4972 card and 4H-SiC phase matched by ICSD 98-016-4971 card. Diffraction pattern on 46S also showed lattice parameter, composition and crystallite size changes. The lattice parameter changes not significant. For 6S and 46S samples at 1400 ^{o}C, the 6H-SiC phase changes into other phases more than 50 % from its original weight percentage.

Keywords: silicon carbide, 4H-SiC, 6H-SiC, oxidation, high temperature.

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ABSTRAK

PERUBAHAN FASA 4H DAN 6H SIC YANG TEROKSIDASI PADA TEMPERATUR TINGGI*. Telah dilakukan proses oksidasi pada silikon karbida yang mengadung fasa 6H dan silikon karbida yang mengandung fasa 4H dan 6H. Silikon karbida merupakan keramik non oksida dengan sifat-sifat unggulnya yang sangat potensial digunakan dalam dunia industri. Dalam industri nuklir silikon karbida digunakan sebagai bahan struktur kelongsong pada bahan bakar reaktor air ringan light water reactor (LWR) dan sebagai pelapis pada kernel bahan bakar reaktor gas temperatur tinggi (RGTT). Pada studi ini dilakukan simulasi oksidasi silikon karbida pada kernel apabila terjadi kegagalan pada pipa pendingin utamanya. Sampel dibentuk dari serbuk silikon karbida yang di pres hingga berbentuk pelet dengan diameter 12,7 mm dan ketebalan 1.0 mm kemudian dioksidasi pada temperatur 1000 ^{o}C, 1200 ^{o}C dan 1400 ^{o}C selama 1 jam. Sampel sebelum dan setelah dioksidasi dilakukan penimbangan dan pengujian difraksi sinar-X menggunakan Difraktometer Panalytical Empyrean dengan Cu sebagai sumber sinar-X. Analisis pola difraksi dilakukan menggunakan aplikasi General Structure Analysis System (GSAS), dengan hasil yang diperoleh adalah perubahan parameter kisi dan kandungan fasa SiC-nya. Hasil percobaan menunjukkan bahwa semua sampel yang teroksidasi mengalami peningkatan berat. Oksidasi sampel 6S menyebabkan kenaikan berat tertinggi pada temperatur 1200 ^{o}C, sedangkan sampel 46S memiliki berat dengan kecenderungan meningkat seiring dengan meningkatnya temperatur oksidasi. Analisis pola difraksi sinar-X menunjukkan bahwa fasa dominan yang terbentuk pada sampel 6S adalah fasa 6H-SiC yang didekati dengan model dari kartu ICSD 98-001-5325. Pola difraksi sampel 6S menunjukkan adanya perubahan parameter kisi, perubahan komposisi dan perubahan ukuran kristalitnya. Perubahan panjang kisi memiliki kencenderungan berkurang dari nilai model dan sebelum dioksidasi, sedangkan komposisi silikon karbida paling rendah atau yang paling banyak terkonversi menjadi fasa lain mencapai 66.85 %, yang terjadi pada temperatur oksidasi 1200 ^{o}C. Sampel 46S mengandung fasa 4H-SiC dan 6H-SiC. Fasa 6H-SiC didekati dengan model dari kartu ICSD 98-016-4972 dan fasa 4H-SiC didekati dengan model dari kartu ICSD 98-016-4971. Pola difraksi sampel 46S menunjukkan adanya perubahan parameter kisi, perubahan komposisi dan perubahan ukuran kristalitnya. Perubahan panjang kisi pada sampel 46S tidak terlalu signifikan. Fasa 6H-SiC pada sampel 6S dan 46S dengan temperatur oksidasi 1400 ^{o}C mengalami perubahan menjadi fasa oksida dan lainnya sebesar lebih dari 50 % persen berat awalnya.*

Kata kunci*: silikon karbida, 4H-SiC, 6H-SiC, oksidasi, temperatur tinggi.*

INTRODUCTION

Silicon carbide (SiC) is ceramic material with properties high hardness, low bulk density and high oxidation resistance which make SiC suitable for a wide range of industrial applications [1]. SiC widely used as refractory material [2] that also interest in using as the nuclear fuel cladding material in light water reactor (LWR) [3,4] and as coating layer in high temperature gas cooled reactor (HTGR) fuel [5]. Sintering of silicon carbide was first performed by Prochazka by using boron and carbon through a solid state mechanism that performed at 2150-2200 ^{o}C [1]. Silicon carbide in nature can be found as extremely rare mineral called moissanite [2]. It was first discovered by H. Moissan on meteorite located in the Diablo Canyon of Arizona in 1893. In 1891, E. G. Acheson synthesized silicon carbide in the laboratory and named as 'Carborundum' [2]. SiC has two major polytypes, which are alpha SiC (α-SiC) having hexagonal crystal structure (similar to Wurtzite) and the beta modification (β-SiC), with a zinc blende crystal structure (similar to diamond) [2]. SiC exits in various crystallographic phases with more than 170 polytypes. Despite lower carrier mobilities compared to β-SiC, hexagonal 4H and 6H SiC polytypes are the most commonly used for devices because of good quality crystal wafer availability and larger bandgap.

SiC is a non-oxide and it has the tendency to get oxidized in presence of oxygen or oxidizing agents[2]. Oxidation of SiC based materials is also an important issue. The oxidation of SiC-based material generally can be different into two oxidation regimes: active and passive oxidation [2,4]. The oxidation behavior depends on the oxidizing conditions such as the oxidizing species, temperature, total pressure, and partial pressure of the oxidant[2,4,6]. The particle size also strongly influences the kinetics oxidation, where the weight gain and rate constant increasing with decreasing the SiC particle size [7]. In active oxidation, at low oxygen partial pressures and high temperatures, non-protective oxide film formation is expected as a result of significant SiO vapor formation:

$$SiC\ (s) + O_2\ (g) \rightarrow SiO(g) + CO\ (g) \qquad (1)$$

SiO_2 formed gets vaporized after its formation leading to loss of mass. In contrast, passive oxidation occurs at high oxygen partial pressures and very high temperatures. In passive oxidation, a protective SiO_2 layers forms according to:

$$SiC\ (s) + 3/2\ O_2\ (g) \rightarrow SiO_2\ (s) + CO(g) \qquad (2)$$

SiO_2 forms during passive oxidation get deposited over the surface of SiC leading to net increase in the mass. It was observed that SiO_2 formed at lower temperature develops layer over the surface of SiC and protects it from further oxidation to SiO occurred at higher temperatures. These protective action continues up to the melting point of SiO_2 at 1996 K [2]. Hijikata [8] proposed Si and C emission model for mechanism SiC oxidation. Figure 1, illustrated the Si and C emission model for SiC oxidation. Considering Si and C atoms emitted from the interface during the oxidation as well as the oxidation process of C. X is denote as the oxide thickness. In this model the oxide growth rate well reproduces over the entire thickness range for both the C and Si faces. Describing the oxide growth process in SiC needed the oxidation and emission of C and the emission Si take into account. In case the absence of interstitial accumulation and Si emission rate might be contributing to the large difference in oxide growth rate between the polar faces[9]. A face terminated oxidation behavior on 4H-SiC has been observed which indicates that the oxidation growth rate on C-face is faster than on Si face[10]. The oxide layer thickness increased slowly below 1200 ^{o}C and rapidly when the temperatures are higher than 1500 ^{o}C[11]. This term oxidation process can use as film growth on SiC surface that applicable in electronic devices[12].

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SiC in kernel nuclear fuel is provide a crucial safety in this nuclear system. SiC layer is considered the most importance since not only provides the TRISO particle with structural integrity but also it is the primary layer for containing radioactive fission products at elevated temperatures. Test on safety have been carried out to study the performance of this SiC coating layer. Result revealed several potential failure mechanisms of the SiC layer, which included pressure vessel failure caused by internal gas pressure and irradiation stress, chemical corrosion and thermal decomposition at very high temperatures. Many postulated accidents must also be taken into account in evaluating the reliability of the SiC layer. Air ingress is among the postulated accidents in HTGR. Under normal operation conditions of an HTGR reactor, the fuels work in helium loops and are isolated from air by several protective barriers. However postulated accidents in which a primary coolant pipe is ruptured, air invades the reactor core. The fuel sphere might be exposed to air [11]. Furthermore, the oxidation results obtained can also provide information of SiC phase changes during oxidation.

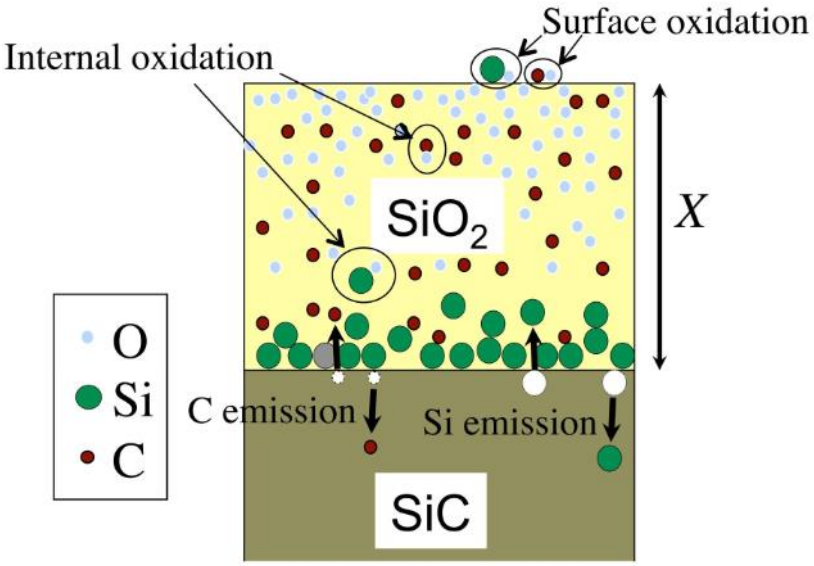

Figure 1. Schematic mechanism SiC oxidation[8]

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provide information of SiC phase changes during oxidation.

In this study, investigation and comparison of the phase changes on the SiC in which containing only the 6H polytype with combining polytype silicon carbide the 4H- and 6H- polytypes relating to the high temperature oxidation has been carried out. The samples were oxidized at temperature 1000 ^{o}C, 1200 ^{o}C, and 1400 ^{o}C for 1 hour and the tests were conducted in air atmosphere. The weight changes on samples were analyzed by analytical balance. As a passive oxidation occur in this experiment, the sample showed weight gain after oxidation [2,4]. The phase changes of the SiC samples were observed by the X-ray diffractometer (XRD). The relationship between the lattice parameter of SiC phase and weight changes was discussed.

EXPERIMENTAL PROCEDURE

The material used in this study were polycrystalline SiC. The samples were prepared in pellet form, with dimensions of approximately diameter 12.7 mm and thickness 1.0 mm. Sample with phase only 6H-SiC coded by 6S and sample containing 4H-SiC and 6H-SiC coded by 46S. A heating furnace of thermolyne was used to oxidation the samples. All of tests were conducted with heating rate at 10 ^{o}C/min to the desired temperature achieved. The samples were oxi-dized for 1 hour in the air atmosphere and the cooling rate at 5 ^{o}C/min until reached the room temperature while the samples still inside the furnace chamber. The oxidation temperatures were conducted at 1000 ^{o}C, 1200 ^{o}C and 1400 ^{o}C in air atmosphere. Analytical balance was used to weighted the samples before and after the oxidation. In order to observe the structural change on the samples by X-ray diffraction, the Panalytical Empyrean diffractometer with The Cu X-ray source was used. During the X-ray Diffraction process the X-ray source tension parameter was set at 40 kV and current parameter was set at 30 mA with time per step at 1 s. The analysis of the diffraction pattern was performed by the General Structure Analysis System (GSAS) software.

RESULT AND DISCUSSION

Results from the analytical balance of the samples weight changes represented at Figure 2. The sample showed weight gain after oxidation as reported by [2,4]. These weight values were combination of the SiC phase(s) and the other phases that formed during oxidation. The 6S oxidation weight changes percentage at 1000 ^{o}C was 4.55 %, and the change continues increased. The higher percentage was the oxidation at 1200 ^{o}C, where the changes reached 24.44 %. The samples oxidation for 46S, showed lower oxidation weight changes percentage. The higher percentage was occurred at 1400 ^{o}C, with percentage reached 5.30 %. The next section will show the phase that affected the weight changes.

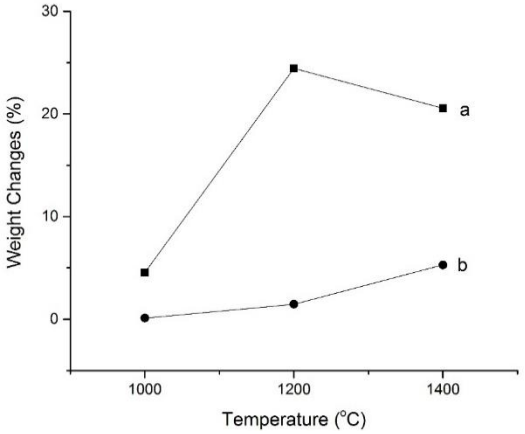

Figure 2. Weight changes of (a) 6S and (b) 46S during oxidation under air atmosphere

a. Silicon carbide containing 6H polytype (6H-SiC or 6S)

The diffraction pattern in Figure 3 showed for samples 6S and the oxidation samples. The oxidation diffraction patterns showed another phases formed beside the 6H-SiC phase. During the oxidation, there

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were interaction induced by heat between oxygen with silicon carbide while temperature increase. The diffraction pattern showed peak shifted and there some broadening peaks. Shifted peaks indicating there were lattice parameter changes and the broadening peaks there were changes in crystallite size. The original lattice parameters and the changes for 6H-SiC phases were tabulated in Table 1. Some peaks broadening tabulated in Table 2.

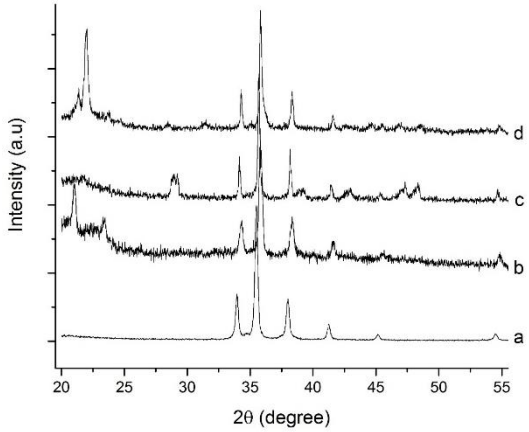

Figure 3. Diffraction patterns of SiC 6H polytype, (a) without oxidation (as received) and oxidation at (b) 1000 ^{o}C, (c) 1200 ^{o}C, and (d) 1400 ^{o}C under air atmosphere.

The original phases in the 6S was mossainate 6H that matched to the card 98-001-5325 from ICSD data base. These refined parameters were analyzed by GSAS software using the Rietveld method [13-14]. The original model for analyzed all the samples for 6S was based on the ICSD card 98-001-5325 [15]. The matched phases on the oxidation samples not only 6H-SiC appear, there were other several crystalline phases form the oxygen-silicon carbide interaction such as silicon dioxide. The other phases not for our concerns now.

The Rietveld method results for 6S showed changes in a-axis and c-axis values. The as received SiC had higher a-axis and c-axis values compared to the model. The lattice parameters all of oxidation samples changes compared the as received SiC. The lattice parameters values showed decreasing at 1000 ^{o}C, and then increasing again for 1200 ^{o}C and 1400 ^{o}C, but the values still lower than the as received SiC. The lower lattice parameters values for 6S indicating had smaller volume and possibility had higher density. The 6H-SiC phase weight changes in the oxidation samples was higher at the oxidation temperature 1200 ^{o}C. In Figure 2, the weight changes percentage for 6S showed higher changes at 1200 ^{o}C in this case correlate to the weight percentage for other phases, which is the higher weight percentage value about 66.85 %. The 6S than contain only 6H-SiC indi-cate the oxidation at high temperature its weight changes were significantly influenced by other phases that formed during oxidation. The oxide phases were formed slowly below 1200 ^{o}C [11]. At 1400 ^{o}C the oxide layer in the surface of SiC already reached saturation where there no more emission of Si and C atoms from the surface of SiC or internal of oxide layer. Peaks detection in as received SiC for the three peaks with higher intensities were tabulated in Table 2.

Table 1. Phase contents in the 6S

Parameter	Model	w/o oxidation	1000 ^{o}C	1200 ^{o}C	1400 ^{o}C
Moissanite 6H 98-001-5325[15]					
Crystal system:	Hexagonal	Hexagonal	Hexagonal	Hexagonal	Hexagonal
Space group:	P 63 m c	P 63 m c	P 63 m c	P 63 m c	P 63 m c
a=b= (Å):	3.0810	3.08360(3)	3.072658(5)	3.07982(6)	3.08073(8)
c=(Å):	15.1170	15.13064(5)	15.08227(7)	15.11628(2)	15.11531(1)
Alpha=Beta(°):	90	90	90	90	90
Gamma (°):	120	120	120	120	120

Wt%:		100.000	74.266	33.152	47.548
Other phases:					
Wt%:			25.743	66.848	52.452

Table 2. Peaks of SiC 6H polytype.

2θ (degree) (w/o oxidation)	SiC Polytype	FWHM			
		w/o oxidation	1000 °C	1200 °C	1400 °C
34.107	6H	0.160	0.240	0.080	0.120
35.574	6H	0.260	0.080	0.140	0.140
59.860	6H	0.200	0.080	0.100	0.100

According to the without oxidation sample the broadening peaks analyzed through the Full Width at Half Maximum (FWHM) value. In the peak at 35.574 degree and 59.860 degree there were decreasing FWHM value in the oxidation at 1000 °C and had similar FWHM value in the oxidation at 1200 °C and 1400 °C. Different to the peak at 34.107 degree that changes to oxidation temperature. This FWHM value changes phenomena occurs for each peaks. These meant the crystallite size changes with the temperature oxidation. At 1000 °C the crystallite size was decreasing compare to the original crystallite size. At 1200 °C the crystallite size was increasing compared at 1000 °C, and at 1400 °C was decreasing but still bigger compared the original crystallite size.

b. Silicon carbide containing 4H and 6H polytype (4H-SiC and 6H-SiC or 46S)

The diffraction pattern changes during oxidation process for 46S showed in Figure 4. The phases changes can be observed by several peaks from SiC 4H polytype and SiC 6H polytype. The diffraction pattern also showed peak shifted as the 6H-SiC and there also some broadening peaks. The original lattice parameters and the changes for 4H-SiC and 6H-SiC phases were tabulated in Table 3. Table 4 showed the corresponding peaks to each of SiC polytype.

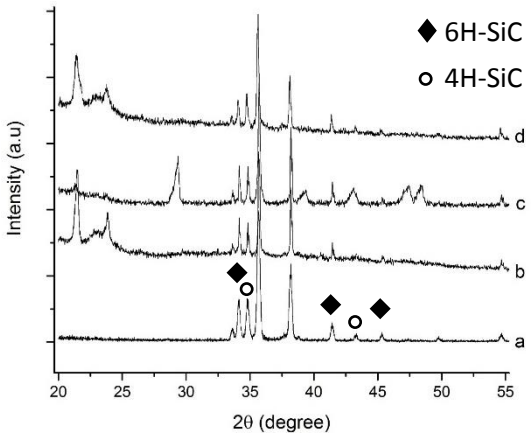

Figure.4..Diffraction patterns of SiC containing 4H and 6H polytype, (a) without oxidation (as received) and oxidation at (b) 1000 °C, (c) 1200 °C, and (d) 1400 °C under air atmosphere. Tick marks indicate non overlapping peak between 4H and 6H.

The original phase of the 4H-silicon carbide was matched to the card 98-016-4971 [15] and the 6H-silicon carbide was matched to the card 98-016-4972 [15], the both cards were taken from ICSD data base. These refined parameters were analyzed by GSAS software using the Rietveld method. The original model for analyzed all the samples were based on the ICSD card 98-016-4972 and 98-016-4971. The matched phases on the oxidation samples not only 4H-SiC and 6H-SiC phases, there were several phases form the oxygen-silicon carbide interaction such as silicon dioxide. These other phases not for our concerns now.

The Rietveld method for these samples also showed changes in a-axis and c-axis value. The as received SiC had lower

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a-axis value but higher for c-axis value compared to the model. The lattice parameters values for 4H-SiC and 6H-SiC changes during the oxidation. For 6H-SiC, only at 1400 ^{o}C had a-axis and c-axis values higher than as received SiC. This indicate the 6H-SiC that oxidated at 1400 ^{o}C had highest volume unit cell that affected to the density value become smaller compare to the as received SiC. For 4H-SiC during oxidation increasing occurred in lattice parameter. This indicate the samples had higher volume that possibility had smaller density compared to the as received SiC. The analysis result for the as received SiC content for 6H-SiC were 62.717 % and 37.283 % for 4H-SiC. The content of each SiC polytype at 1000 ^{o}C was lower compared to the as received SiC. At this temperature, more than 60 % for each polytype SiC converted into other phases. At 1200 ^{o}C, the percentage converted SiC decreasing down to around 37 % for each polytypes of SiC. At the oxidation temperature 1400 ^{o}C, the 6H-SiC in the sample 46S had more the oxygen uptake compared to the 4H-SiC. These state same as the 6H-SiC at the samples 6S, where the converted SiC into other phase were more than 50 % from it's original weight percentage. In Figure 2, the weight changes percentage for 46S showed increasing changes that correlated to the weight percentage for 4H-SiC phase. In the combination of 4H- and 6H-SiC polytype, the 4H phase existence in the samples had the higher contribution into the SiC weight compared to the 6S sample that other phases that containing the role majoring by oxide phase.

Same as to the 6H-SiC samples these 46S samples the broadening peaks analyzed through the Full Width at Half Maximum (FWHM) value. The 6H-SiC and 4H-SiC phase had bigger crystallite size tendency along with increasing the temperature. Crystallite size of 6H-SiC decreasing for peak at 45.330 degree at temperature 1000 ^{o}C and 1400 ^{o}C. While, the 4H-SiC phase happened for peak at 43.289 degree at all oxidation temperature.

Corresponding to the SiC oxidation, the mechanism in passive oxidation was formed a protective layer to inhibit further SiC oxidation. In our finding, at high temperature the 6H-SiC more reactive to the air compared the 4H-SiC. This was showed by the weight percentage of 4H-SiC in 46S samples that relativly low converted into other phases during oxidation compared to the 6H-SiC.

Table 3. Phase contents in the 46S.

Parameter	Model	w/o oxidation	1000 °C	1200 °C	1400 °C
Moissanite 6H 98-016-4972[15]					
Crystal system:	Hexagonal	Hexagonal	Hexagonal	Hexagonal	Hexagonal
Space group:	P 63 m c	P 63 m c	P 63 m c	P 63 m c	P 63 m c
a=b= (Å):	3.0820	3.08129(7)	3.08084(7)	3.08009(1)	3.08143(0)
c=(Å):	15.0920	15.11467(6)	15.12063(5)	15.11809(5)	15.12258(4)
Alpha=Beta(°):	90	90	90	90	90
Gamma (°):	120	120	120	120	120
Wt%:		62.717	22.061	39.492	30.706
Moissanite 4H 98-016-4971[15]					
Crystal system:	Hexagonal	Hexagonal	Hexagonal	Hexagonal	Hexagonal
Space group:	P 63 m c	P 63 m c	P 63 m c	P 63 m c	P 63 m c
a=b= (Å):	3.0820	3.07769(8)	3.08430(2)	3.07975(8)	3.08144(0)
c=(Å):	10.0610	10.07763(2)	10.10144(7)	10.08449(1)	10.08571(1)
Alpha=Beta(°):	90	90	90	90	90
Gamma (°):	120	120	120	120	120
Wt%:		37.283	14.148	23.338	35.738
Other phases:					
Wt%:			63.801	47.170	34.556

Table 4. Peaks of SiC 6H polytype and SiC 4H polytype.

2θ (degree) (w/o oxidation)	SiC Polytype	FWHM			
		w/o oxidation	1000 °C	1200 °C	1400 °C
34.095	6H	0.180	0.060	0.060	0.120
41.390	6H	0.120	0.060	0.060	0.080
45.330	6H	0.100	0.200	0.060	0.160
34.745	4H	0.120	0.060	0.060	0.100
43.289	4H	0.120	0.160	0.160	0.160
57.294	4H	0.320	0.080	0.080	0.080

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The a-axis value for 6H-SiC at room temperature was at 3.0815 Å [16] and 3.0810 Å [17]. For c-axis value was at 15.117 Å [16] and 15.092 Å [17]. According to [16] the a-axis and c-axis were increased as temperature rising. Our results showed a-axis and c-axis value for 6H-SiC were around these values. For 4H-SiC, at room temperature was at 3.079 Å, 3.090 Å[18] and 3.081 Å[17]. For c-axis value was 10.053 Å [18] and 10.061 Å [17]. Our results for a-axis value were not significanly different, but our findings for the c-axis values slightly higher than reported by [17,18]. SiC layer performance corelate to the kernel nuclear fuel must be done by another testing such as mechanical and/or corrosion test. In this study only show how many weight the SiC converted into other phases during oxidation. That we know the change of phases, would changes the surface properties such as its chemical, mechanical and corrosion properties.

CONCLUSIONS

Two samples of SiC successfully oxidized at high temperature in air atmosphere. The samples undergo to weight gain during oxidation. Analysis on diffraction pattern using Rietveld method in GSAS software, capable to determine the weight percentage of phases and the lattice parameter changes. For the 6S showed the highest weight changes at 1200 ^{o}C, this was correlated to the amount of 6H-SiC phase that converted into the other phases. The peak shifted and broadening were clearly occurred in the diffraction pattern. The peak shifted tendency in samples 46S similar to the lattice parameter value between the as received SiC and the oxidation samples. However, for peak broadening tendency to had bigger crystallite size. The 6H-SiC showed highly oxidated compare to the 4H-SiC at this range temperature. Oxidation at 1400 ^{o}C showed the 6H-SiC in both samples converted into other phases more than 50 % of weight percentage.

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